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JC657 U.S. PTO 11/16/00

UTILITY PATENT APPLICATION TRANSMITTAL

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Attorney Docket No.	042390.P8276
First Inventor	Anne E. Miller
Title	SLURRY AND METHOD FOR CHEMICAL MECHANICAL POLISHING OF COPPER
Express Mail Label No.	EL034435664US

APPLICATION ELEMENTS

See MPEP chapter 600 concerning utility patent application contents

ADDRESS TO: Assistant Commissioner for Patents
Box Patent Application
Washington, DC 20231

1. ☒ Fee Transmittal Form (e.g., PTO/SB/17) *(Submit an original and a duplicate for fee processing)*
2. ☐ Applicant claims small entity status. See 37 CFR 1.27.
3. ☒ Specification [Total Pages 20]
   (preferred arrangement set forth below)
   - Descriptive title of the Invention
   - Cross References to Related Applications
   - Statement Regarding Fed sponsored R & D
   - Reference to sequence listing, a table, or a computer program listing appendix
   - Background of the Invention
   - Brief Summary of the Invention
   - Brief Description of the Drawings *(if filed)*
   - Detailed Description
   - Claim(s)
   - Abstract of the Disclosure
4. ☒ Drawing(s) *(35 U.S.C. 113)* [Total Sheets 4]
5. Oath or Declaration [Total Pages 4]
   a. ☐ Newly executed (original or copy)
   b. ☐ Copy from a prior application (37 C.F.R. § 1.63(d)) *(for continuation/divisional with Box 18 completed)*
      i. ☐ DELETION OF INVENTOR(S)
      Signed statement attached deleting inventor(s) named in the prior application, see 37 CFR 1.63(d)(2) and 1.33(b)
6. ☐ Application Data Sheet. See 37 CFR 1.76
7. ☐ CD-ROM or CD-R in duplicate, large table or Computer Program *(Appendix)*
8. Nucleotide and/or Amino Acid Sequence Submission *(if applicable, all necessary)*
   a. ☐ Computer Readable Form (CRF)
   b. Specification Sequence Listing on:
      i. ☐ CD-ROM or CD-R (2 copies); or
      ii. ☐ paper
   c. ☐ Statements verifying identity of above copies

JC675 U.S. PTO 09/715282 11/16/00

ACCOMPANYING APPLICATION PARTS

9. ☐ Assignment Papers (cover sheet & document(s))
10. ☐ 37 C.F.R. § 3.73(b) Statement *(when there is an assignee)* ☐ Power of Attorney
11. ☐ English Translation Document *(if applicable)*
12. ☐ Information Disclosure Statement (IDS)/PTO-1449 ☐ Copies of IDS Citations
13. ☐ Preliminary Amendment
14. ☐ Return Receipt Postcard (MPEP 503) *(Should be specifically itemized)*
15. ☐ Certified Copy of Priority Document(s) *(if foreign priority is claimed)*
16. ☐ Request and Certification under 35 U.S.C. 122 (b)(2)(B)(i). Applicant must attach form PTO/SB/35 or its equivalent.
17. ☐ Other: ....

18. If a CONTINUING APPLICATION, *check appropriate box, and supply the requisite information below and in a preliminary amendment:*
☐ Continuation ☐ Divisional ☐ Continuation-in-part (CIP) of prior application No: ________

Prior application Information: Examiner________ Group/Art Unit: ________

For **CONTINUATION OR DIVISIONAL APPS only: The entire disclosure of the prior application, from which an oath or declaration is supplied under Box 5b, is considered a part of the disclosure of the accompanying continuation or divisional application and is hereby incorporated by reference. The incorporation can only be relied upon when a portion has been inadvertently omitted from the submitted application parts.**

18. CORRESPONDENCE ADDRESS

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Signature	Donna Jo Coningsby	Date	11/16/00

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FEE TRANSMITTAL for FY 2000

Patent fees are subject to annual revision

TOTAL AMOUNT OF PAYMENT ($) 1,156.00

Complete if Known	
Application Number	
Filing Date	November 16, 2000
First Named Inventor	Anne E. Miller
Examiner Name	
Group/Art Unit	
Attorney Docket No.	042390.P8276

METHOD OF PAYMENT (check one)

1. ☐ The Commissioner is hereby authorized to charge indicated fees and credit any overpayments to:

Deposit Account Number: 02-2666

Deposit Account Name: Blakely, Sokoloff, Taylor & Zafman LLP

☒ Charge Any Additional Fee(s) Required Under 37 CFR §§ 1.16, 1.17, 1.18 and 1.20.

☐ Applicant claims small entity status See 37 CFR 1.27.

2. ☒ Payment Enclosed:

☒ Check ☐ Credit card ☐ Money Order ☐ Other

FEE CALCULATION

BASIC FILING FEE

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description	Fee Paid
1	710	201	355	Utility filing fee	710.00
6	320	206	160	Design filing fee	
7	490	207	245	Plant filing fee	
8	710	208	355	Reissue filing fee	
4	150	214	75	Provisional filing fee	

SUBTOTAL (1) ($) 710.00

EXTRA CLAIM FEES

			Extra Claims		Fee from below		
al Claims	27	- 20** =	7	X	18.00	=	$126.00
ependent iims	7	- 3** =	4	X	80.00	=	$320.00
iltiple Dependent						=	

**or number previously paid, if greater, For Reissues, see below

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description
103	18	203	9	Claims in excess of 20
102	80	202	40	Independent claims in excess of 3
104	260	204	135	Multiple Dependent claim, if not paid
109	80	209	40	**Reissue independent claims over original patent
110	18	210	9	**Reissue claims in excess of 20 and over original patent

SUBTOTAL (2) ($) 446.00

FEE CALCULATION (continued)

3. **ADDITIONAL FEE**

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description	Fee Paid
105	130	205	65	Surcharge - late filing fee or oath	
127	50	227	25	Surcharge - late provisional filing fee or cover sheet.	
139	130	139	130	Non-English specification	
147	2,520	147	2,520	For filing a request for reexamination	
112	920*	112	920*	Requesting publication of SIR prior to Examiner action	
113	1,840*	113	1,840*	Requesting publication of SIR after Examiner action	
115	110	215	55	Extension for response within first month	
116	390	216	195	Extension for response within second month	
117	890	217	445	Extension for response within third month	
118	1,390	218	695	Extension for response within fourth month	
128	1,890	228	945	Extension for response within fifth month	
119	310	219	155	Notice of Appeal	
120	310	220	155	Filing a brief in support of an appeal	
121	270	221	135	Request for oral hearing	
138	1,510	138	1,510	Petition to institute a public use proceeding	
140	110	240	55	Petition to revive - unavoidable	
141	1,240	241	620	Petition to revive - unintentional	
142	1,240	242	620	Utility issue fee (or reissue)	
143	440	243	220	Design issue fee	
144	600	244	300	Plant issue fee	
122	130	122	130	Petitions to the Commissioner	
123	130	123	130	Petitions related to provisional applications	
126	180	126	180	Submission of Information Disclosure Stmt	
581	40	581	40	Recording each patent assignment per property (times number of properties)	
146	710	246	355	Filing a submission after final rejection (37 CFR § 1.129(a))	
149	710	249	355	For each additional invention to be examined (37 CFR § 1.129(b))	
179	710	126	355	Request for Continued Examination (RCE)	
169	900	169	900	Request for expedited examination of a design application	
Other fee (specify)					
Other fee (specify)					

* Reduced by Basic Filing Fee Paid

SUBTOTAL (3) ($)

SUBMITTED BY

Complete (if applicable)

Name (Print/Type)	Donna Jo Coningsby	Registration No. (Attorney/Agent)	41,684	Telephone	(503) 684-6200
Signature	Donna Jo Coningsby			Date	11/16/00

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P8276 PATENT

SLURRY AND METHOD FOR CHEMICAL MECHANICAL POLISHING OF COPPER

Inventors: Anne E. Miller
A. Daniel Feller
Kenneth C. Cadien

"Express Mail" mailing label number EL034435664US

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SLURRY AND METHOD FOR CHEMICAL MECHANICAL POLISHING OF COPPER

Inventors: Anne E. Miller
A. Daniel Feller
Kenneth C. Cadien

Background of the Invention

Field of the Invention

The present invention relates generally to the field of chemical mechanical polishing (CMP), and more specifically, to methods and chemistries for providing increased metal polish rates.

Background

Advances in semiconductor manufacturing technology have led to the development of integrated circuits having multiple levels of interconnect. In such an integrated circuit, patterned conductive material on one interconnect level is electrically insulated from patterned conductive material on another interconnect level by films of material such as, for example, silicon dioxide. These conductive materials are typically a metal or metal alloy. Connections between the conductive material at the various interconnect levels are made by forming openings in the insulating layers and providing an electrically conductive structure such that the patterned conductive material from different interconnect levels are brought into electrical contact with each other. These electrically conductive structures are often referred to as contacts or vias.

Other advances in semiconductor manufacturing technology have lead to the integration of millions of transistors, each capable of switching at high speed. A consequence of incorporating so many fast switching transistors into an integrated circuit is an increase in power consumption during operation. One technique for increasing speed while reducing power consumption is to replace the traditional aluminum and aluminum alloy interconnects found on integrated circuits with a metal such as copper, which offers lower electrical resistance. Those skilled in the electrical arts will appreciate that by reducing resistance, electrical signals may propagate more quickly through the interconnect pathways on an integrated circuit. Furthermore, because the resistance of copper is significantly less than that of aluminum, the cross-sectional area of a copper interconnect line, as compared to an aluminum interconnect line, may be made smaller without incurring increased signal propagation delays based on the resistance of the interconnect. Additionally, because the capacitance between two electrical nodes is a function of the overlap area between those nodes, using a smaller copper interconnect line results in a decrease in parasitic capacitance. In this way, replacing aluminum based interconnects with copper based interconnects provides, depending on the dimensions chosen, reduced resistance, reduced capacitance, or both.

As noted above, copper has electrical advantages, such as lower resistance per cross-sectional area, the ability to provide for reduced parasitic capacitance, and greater immunity to electromigration. For all these reasons, manufacturers of integrated circuits find it desirable to include copper in their products.

While advantageous electrically, copper is difficult to integrate into the process of making integrated circuits. As is known in this field, copper can

adversely affect the performance of metal oxide semiconductor (MOS) field effect transistors (FETs) if the copper is allowed to migrate, or diffuse, into the transistor areas of an integrated circuit. Therefore copper diffusion barriers must be used to isolate copper metal from those transistor areas. Additionally, unlike aluminum based metal interconnect systems which are formed by subtractive etch processes, copper interconnects are typically formed by damascene metal processes. Such processes are also sometimes referred to as inlaid metal processes. In a damascene process, trenches are formed in a first layer, and a metal layer is formed over the first layer including the trenches. Excess metal is then polished off, leaving individual interconnect lines in the trenches. The removal of excess copper is typically accomplished by chemical mechanical polishing. Although there are many known variations of the damascene method of metallization, the most common method for removing the excess copper is by CMP.

Accordingly, there is a need for CMP methods, materials, and apparatus to polish conductive materials such as copper.

Brief Description of the Drawings

Fig. 1 is a schematic cross-sectional view of a copper damascene structure. This structure represents a post-plating, pre-polishing state of fabrication.

Fig. 2 is a flowchart showing the operations in a process of forming a slurry in accordance with the present invention

Fig. 3 is a flowchart showing the operations in a process of polishing a thin film in accordance with the present invention.

Fig. 4 is a flowchart showing the operations in a process of polishing a thin film in accordance with the present invention.

Detailed Description

Methods and slurries for the chemical-mechanical polishing of copper are described. In the following description numerous specific details are set forth to provide an understanding of the present invention. It will be apparent, however, to those skilled in the art and having the benefit of this disclosure, that the present invention may be practiced with apparatus and processes that vary from those of the illustrative examples provided herein.

Terminology

The terms, chip, integrated circuit, monolithic device, semiconductor device or component, microelectronic device or component, and similar terms and expressions, are often used interchangeably in this field. The present invention is applicable to all the above as they are generally understood in the field.

RPM (also rpm) refers to revolutions per minute.

Reference herein to “one embodiment”, “an embodiment”, or similar formulations, means that a particular feature, structure, or characteristic described in connection with the embodiment is included in at least one embodiment of the present invention. Thus, the appearances of such phrases or formulations herein are not necessarily all referring to the same embodiment. Furthermore, various particular features, structures, or characteristics may be combined in any suitable manner in one or more embodiments.

Overview

Polishing of copper metal layers in connection with the formation of conductive interconnect lines for integrated circuits is becoming more important for the semiconductor industry. Unlike aluminum metallization, which is typically formed on integrated circuits by subtractive metal etch, copper interconnect lines are typically formed by way of a damascene, or inlaid, metal process. Such a process requires the removal, typically by chemical mechanical polishing, of the excess copper.

Several prior art slurries for chemical mechanical polishing of copper have had problems associated with them. For example, one such prior art slurry, based on a hard abrasive such as Al_2O_3, tended to cause excessive scratching and had an unpleasant odor. In another prior art example, a copper polish slurry contained propianic acid and a silica abrasive but had unsatisfactory characteristics with respect to corrosion, scratching, and odor.

An exemplary copper polish slurry, in accordance with the present invention, may be formed by combining a chelating, organic acid buffer system such as citric acid and potassium citrate; and an abrasive, such as for example colloidal silica and an oxidizer, such as hydrogen peroxide (H_2O_2) Alternative copper polish slurries, in accordance with the present invention, may be formed by further combining a corrosion inhibitor such as benzotriazole (BTA).

Advantageous properties of slurries in accordance with the present invention include the enhancement of Cu removal rates to >3000 angstroms per minute. Additionally, this high polish rate is achieved while maintaining local pH stability and substantially reducing global and local corrosion as compared to prior art copper polish slurries. Those skilled in the art will

appreciate that local pH stability provides for reduced within-wafer non-uniformity and reduced corrosion defects.

The Slurry

Slurries, in accordance with the present invention, include a buffer system to increase the polish rate of a metal CMP system. These slurries are formed by combining a chelating organic acid buffer system such as citric acid and potassium citrate, with an abrasive such as colloidal silica. If the metal to be polished is copper or a copper alloy, then an oxidizer such as hydrogen peroxide should be combined with the slurry mixture. It will be appreciated by those skilled in the art that combining such ingredients may be done in any appropriate container, and may include mixing. Furthermore these ingredients may be combined outside of a container, such as, for example on a polishing pad. Alternative inventive slurries may be formed by further combining the above with a corrosion inhibitor such as benzotriazole. Such slurries are particularly useful for polishing copper, and copper diffusion barriers.

An exemplary slurry, in accordance with the present invention, for chemical mechanical polishing, has a pH of approximately 3.8, and includes a SiO_2 abrasive, a H_2O_2 oxidizer, a benzotriazole corrosion inhibitor, and a citric acid/potassium citrate buffer system. These ingredients are combined, typically with water, to form the slurry. Those skilled in the art will appreciate that the slurry is a mixture of these ingredients, that various chemical reactions may occur amongst the ingredients, and that the slurry may contain various mixture and reaction products of the ingredients, including, but not limited to, complexes and disassociated ionic species. In other words, the slurry that results from combining, or mixing the ingredients, will contain at

equilibrium, or at such other conditions as it may be subjected to, chemical constituents that arise by virtue of the combination of the ingredients in accordance with the present invention. It is noted that slurries in accordance with the present invention may have a pH in the range of 3 to 6.

In one particular illustrative slurry, the citric acid/potassium citrate buffer system is provided by including in the slurry mixture approximately 3 g/l of citric acid and approximately 3 g/l of potassium citrate.

An abrasive suitable for use in the embodiments of the present invention is a precipitated SiO_2. Precipitated SiO_2 is sometimes referred to in this industry as colloidal, although this term, i.e., colloidal, is not a technically accurate designation for this material. The illustrative slurry may contain 5 wt.% silica such as Klebesol 1498-50 (available from Rodel, Inc., 3804 East Watkins Street, Phoenix, AZ 85034).

The illustrative slurry may further be formed from combining hydrogen peroxide with the slurry mixture such that this oxidizer comprises 3 wt.%. Benzotriazole may be combined with the slurry mixture as the corrosion inhibitor. In the illustrative embodiment, the slurry mixture includes 0.015M benzotriazole.

Method

In an embodiment of the present invention, a copper damascene structure is polished to form individual interconnects. Fig. 1 shows a copper damascene structure prior to the removal of the excess copper and copper diffusion barrier layer. An interlayer dielectric (ILD) layer is patterned to form ILD **102** on a surface of a wafer as illustrated in the figure. ILD **102** has a thickness represented by T_{ILD} in Fig. 1. A copper diffusion barrier **104** is formed over the exposed surfaces of the wafer and ILD **102**. Various

materials may be used as the copper diffusion barrier. Tantalum and tantalum nitride may each be used as copper diffusion barriers. Typically, a copper seed layer is then formed on copper diffusion barrier **104**. A complete copper layer **106** is then formed, typically by plating, over diffusion barrier **104**. That portion of the copper that is above the top surface **103** of ILD **102** is considered to be excess. It can be seen by inspection of Fig. 1 that removal of the excess copper will result in the formation of two separate conductive interconnect structures.

An embodiment of the process of forming a slurry in accordance with the present invention is illustrated in the flow diagram of Fig. 2.

As shown in block **202** of Fig. 2, a chelating organic acid buffer system and an abrasive are combined with water. In one embodiment the chelating organic acid buffer system is citric acid and potassium citrate, and the abrasive is colloidal silica. In block **204** an oxidizer is combined with the previously described mixture. In one embodiment the oxidizer is a low electrochemical potential oxidizer such as hydrogen peroxide. In block **206** a corrosion inhibitor is combined with the other ingredients identified above. It will be understood by those skilled in the art that specific order of introducing the ingredients to the slurry mixture may be changed consistent with the present invention. The present invention is not limited in terms of the order of combining ingredients. For example, water and benzotriazole may be combined, then the chelating buffer added, followed by an abrasive, and an oxidizer.

An embodiment of the method of polishing a thin film on a wafer, in accordance with the present invention, is described in conjunction with Fig. 3.

As is well known, in a typical CMP system, a wafer is placed face down on a rotating table covered with a polishing pad, which has been

coated with a slurry. A carrier, which may be attached to a rotatable shaft, is used to apply a downward force against the backside of the wafer. A retaining ring may be used to center the wafer onto the carrier and to prevent the wafer from slipping laterally. By applying the downward force, and rotating the wafer, while simultaneously rotating a pad having slurry thereon, a desired amount of material may be removed from the surface of a thin film.

Fig. 3 shows a flow diagram of a process embodying the present invention. At block **302**, a slurry, having a chelating organic acid buffer system in accordance with the present invention, is prepared, delivered to, and dispensed onto, a polishing pad. The slurry, as described above, may have a pH of approximately 3.8. Then, as shown at block **304**, a wafer with a copper damascene structure formed thereon, is brought into contact with the polishing pad. As shown at block **306** the copper damascene structure is polished. Typical polishing conditions using an orbital polisher (e.g., IPEC 576 Orbital Polisher from Speed-Fam IPEC, 305 North 54th Street, Chandler, AZ 85226) are a down force of approximately 3.75 psi, a spindle speed of approximately 310 rpm, a wafer rotational speed of approximately 19 rpm, a slurry flow rate of approximately 130 ccm, and a delta P of 0.0 psi. Delta P is the pressure difference exerted on the top and bottom of the wafer and allows fine control of the rate at the edge of the wafer. Stacked polishing pads such as the IC1000, with a Suba-4 sub-pad, both made by Rodel, Inc. of 3804 East Watkins Street, Phoenix, AZ 85034, may be used with the slurry to polish copper films. Other commercially available polishing pads may be used with the present invention, for example FX-9 pads available from Freudenberg of Lowell, Massachusetts.

Copper diffusion barriers, such as, for example, tantalum or tantalum nitride, are also successfully polished with slurries and polishing conditions in

accordance with the present invention. In particular, by leaving out the oxidizer but including the chelating organic acid buffer system tantalum based copper diffusion barriers can be effectively polished.

A method of forming copper interconnect in accordance with the present invention is described in conjunction with Fig. 4. Referring to Fig. 4, a illustrative method includes forming a copper diffusion barrier layer over a patterned ILD layer (**402**). This ILD layer, patterned so as to have trenches and vias therein, may be produced with any of the conventional methods of forming an ILD for damascene metal processing. ILD layers may include any suitable dielectric material, including but not limited to, silicon oxide, fluorine-dope silicon oxide, carbon-doped silicon oxide, and ILD layers based on materials other than oxides of silicon, such as, but not limited to organic polymers and porous inorganic materials. In the illustrative embodiment of the present invention a tantalum-based copper diffusion barrier is used. Such a barrier layer may be made of tantalum or tantalum nitride. A copper seed layer is then formed over the copper diffusion barrier layer (**404**). Subsequently, a copper layer is electroplated over the seed layer (**406**). The excess portion of the copper layer (as described above with reference to Fig. 1) is then removed by chemical mechanical polishing (**408**) with a slurry that includes a chelating organic acid buffer system and a low electrochemical potential oxidizer. Such a slurry may contain a citric acid/potassium citrate chelating organic acid buffer system, along with hydrogen peroxide as the oxidizer. An abrasive such as silica is also included in the slurry. A corrosion inhibitor such as benzotriazole may also be included in the slurry. As the copper layer is removed the underlying diffusion barrier layer becomes exposed. The excess portion of the barrier layer, i.e., that portion over the top surface of the ILD, is then removed (**410**). The slurry chemistry is

modified such that the oxidizer is left out for removing the excess portion of the diffusion barrier layer. In other words, a first slurry formulation is used when beginning to polish the copper layer, but a second slurry formulation, similar to the first except for the presence of the oxidizer, is then dispensed to polish the underlying tantalum-based diffusion barrier layer.

With respect to the illustrative embodiment of Fig. 4, copper polishing and barrier layer polishing may be performed on the same pad or on different pads. In the either scenario, copper is polished until a predetermined end point is reached, either by timing the polish, by detecting a change in CMP motor current, or by any other suitable method. If both layers are to be polished on the same pad, the slurry chemistry is modified either by dispensing a second slurry without the oxidizer, or by simply turning off the oxidizer dispenser if this was being delivered directly to the polishing pad. If each layer is to be polished on separate pads, then when the desired endpoint is detected, the wafer may be moved to a second pad to which the second slurry is delivered.

Conclusion

Embodiments of the present invention provide a slurry suitable for chemical mechanical polishing of metals, such as, for example, copper. Other embodiments of the present invention provide methods for forming conductive interconnect lines in an integrated circuit.

An advantage of some embodiments of the present invention is that the chelating agent enhances the copper removal rate to greater than 3000 angstroms per minute while using a low electrochemical potential oxidizer such as hydrogen peroxide. Compatibility with low electrochemical potential

oxidizers reduces the driving force for pitting and other forms of localized corrosion.

A further advantage of some embodiments of the present invention is that in the presence of a citric acid buffer system, the concentration of benzotriazole can be significantly increased to control the static etch rate (sometimes referred to global corrosion) without shutting down the polish rate.

A still further advantage of some embodiments of the present invention is that the chelating, organic acid buffer enhances the removal rate in the presence of a soft abrasive such as colloidal SiO_2.

A still further advantage of some embodiments of the present invention is that the buffer system substantially ensures local pH uniformity which, in turn, decreases within-wafer non-uniformity, and also reduces local corrosion.

A still further advantage of some embodiments of the present invention is that the chelating agent enhances the removal rate over a wide pH range and can be used at high pH with a high pH buffer system.

A still further advantage of some embodiments of the present invention is that the chelating, organic acid buffer system, as opposed to conventional slurries, has no substantial stability, odor, health, or disposal issues associated therewith.

A still further advantage of some embodiments of the present invention is that the ingredients of the slurry form a cost-effective product.

A still further advantage of some embodiments of the present invention is that an effective slurry for polishing Ta and TaN (i.e., copper diffusion barriers), can be formed by combining the chelating, organic acid buffer

system with an abrasive and a corrosion inhibitor, i.e. the oxidizer component need not be added to the slurry.

It will be apparent to those skilled in the art that a number of variations or modifications may be made to the illustrative embodiments described above. For example, various combinations, slurry pH, slurry delivery rate, pad rotation speed, pad temperature, and so on, may be used within the scope of the present invention.

Other modifications from the specifically described apparatus, slurry, and process will be apparent to those skilled in the art and having the benefit of this disclosure. Accordingly, it is intended that all such modifications and alterations be considered as within the spirit and scope of the invention as defined by the subjoined Claims.

What is claimed is:

1. A method of forming copper interconnect, comprising:

forming a copper diffusion barrier layer in at least a damascene structure;

forming a copper layer over the barrier layer;

removing a portion of the copper layer by chemical mechanical polishing with a slurry comprising a chelating organic acid buffer system, colloidal silica, and an oxidizer.

2. The method of Claim 1, wherein the oxidizer comprises hydrogen peroxide.

3. The method of Claim 2, wherein the chelating organic acid buffer system comprises citric acid and potassium citrate.

4. The method of Claim 3, wherein the slurry further comprises a corrosion inhibitor.

5. The method of Claim 4, wherein the corrosion inhibitor comprises benzotriazole.

6. A method of forming copper interconnect, comprising:

forming a barrier layer over a substrate having at least one trench therein;

forming a copper seed layer on the surface of the barrier layer;

forming a copper layer over the barrier and seed layers;

removing a portion of the copper layer by chemical mechanical polishing with a first slurry comprising a chelating organic acid buffer system, colloidal silica, and an oxidizer; and

removing at least a portion of the barrier layer by chemical mechanical polishing with a second slurry comprising a chelating organic acid buffer system, and colloidal silica;

wherein the second slurry is formed without the oxidizer.

7. The method of Claim 6, wherein the barrier layer comprises tantalum.

8. The method of Claim 7, wherein the chelating organic acid buffer system comprises citric acid and potassium citrate.

9. The method of Claim 8, wherein the oxidizer comprises hydrogen peroxide.

10. The method of Claim 9, wherein the first slurry further comprises a corrosion inhibitor.

11. The method of Claim 10, wherein the first slurry has a pH in the range of 3 to 6, and the corrosion inhibitor comprises benzotriazole.

12. A slurry produced by the process comprising: combining citric acid, potassium citrate, silica, hydrogen peroxide, and benzotriazole.

13. The slurry produced by the process of Claim 12, wherein a concentration of citric acid is approximately 3g/l, a concentration of potassium citrate is approximately 3g/l, a concentration of silica is approximately 5 wt. %, a concentration of hydrogen peroxide is approximately 3 wt. %, and a concentration of benzotriazole is approximately 0.015 molar.

14. The slurry produced by the process of Claim 13, further comprising combining the citric acid, potassium citrate, silica, hydrogen peroxide, and benzotriazole with water.

15. A slurry, comprising:

approximately 3 grams/liter of citric acid;

approximately 3 grams/liter of potassium citrate;

approximately 5 wt.% silica;

approximately 3 wt.% hydrogen peroxide;

approximately 0.015 molar benzotriazole; and

the mixture and reaction products thereof.

16. The slurry of Claim 15, wherein the slurry has a pH in the range of 3 to 6.

17. A slurry formed by the process of combining a organic acid, an organic acid salt; approximately 5 wt.% silica; approximately 3 wt.% hydrogen peroxide; and approximately 0.015 molar benzotriazole.

18. The slurry of Claim 17, wherein the organic acid comprises acetic acid.

19. The slurry of Claim 18, wherein the organic acid salt comprises potassium acetate.

20. The slurry of Claim 17, wherein the organic acid comprises 3 grams/liter of citric acid, and the organic acid salt comprises 3 grams/liter of potassium citrate.

21. A slurry for polishing copper diffusion barriers, comprising:

approximately 3 grams/liter of citric acid;

approximately 3 grams/liter of potassium citrate;

approximately 5 wt.% silica;

approximately 0.015 molar benzotriazole; and

the mixture and reaction products thereof.

22. The slurry of Claim 21, wherein the copper diffusion barriers comprise tantalum.

23. The slurry of Claim 21, wherein the slurry has a pH in the range of 3 to 6.

24. A slurry for polishing barriers comprised of tantalum, comprising: organic acid, an organic acid salt, an abrasive, a corrosion inhibitor, and the mixture and reaction products thereof, and wherein no oxidizer is included.

25. The slurry of Claim 24, wherein the organic acid comprise citric acid.

26. The slurry of Claim 24, wherein the corrosion inhibitor comprises benzotriazole, and wherein the slurry has a pH in the range of 3 to 6.

27. The slurry of Claim 25, wherein the organic acid salt comprises potassium citrate.

ABSTRACT OF THE DISCLOSURE

A copper polish slurry, useful in the manufacture of integrated circuits generally, and for chemical mechanical polishing of copper and copper diffusion barriers particularly, may be formed by combining a chelating, organic acid buffer system such as citric acid and potassium citrate; and an abrasive, such as for example colloidal silica. Alternative copper polish slurries, in accordance with the present invention, may be formed by further combining an oxidizer, such as hydrogen peroxide, and/or a corrosion inhibitor such as benzotriazole. Advantageous properties of slurries in accordance with the present invention include the enhancement of Cu removal rates to >3000 angstroms per minute. This high polish rate is achieved while maintaining local pH stability and substantially reducing global and local corrosion as compared to prior art copper polish slurries. Local pH stability provides for reduced within-wafer non-uniformity and reduced corrosion defects. Furthermore, copper diffusion barriers such as tantalum or tantalum nitride may also be polished with such slurries wherein the oxidizer is not included.

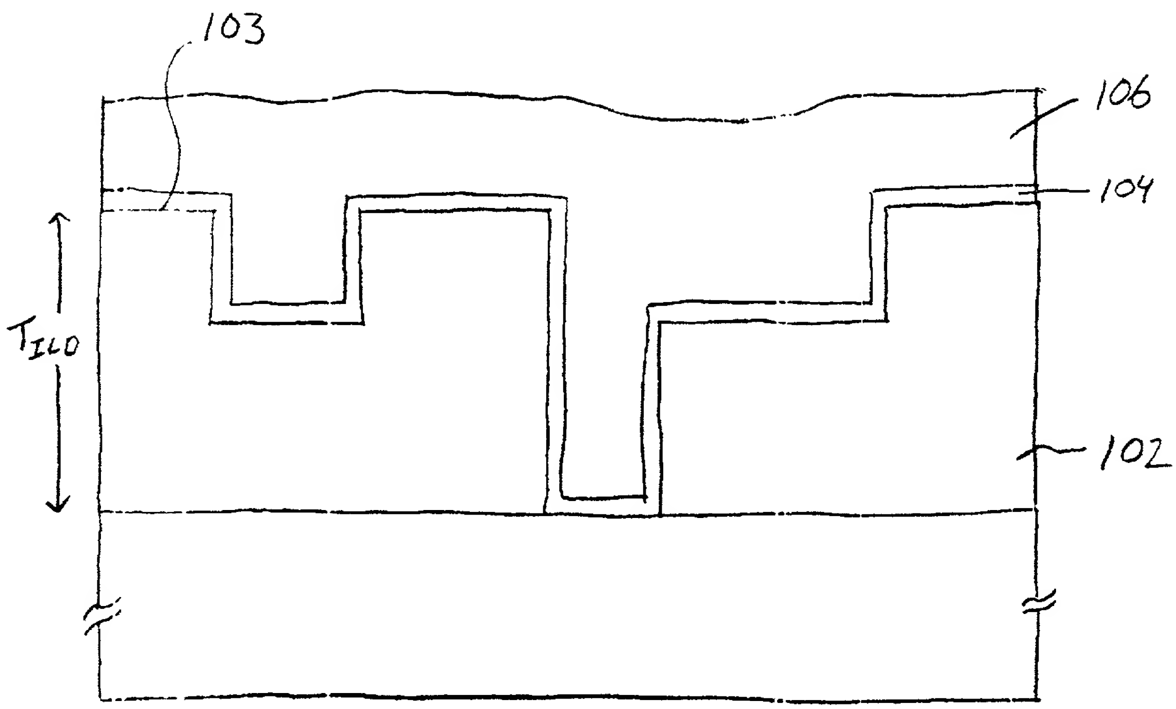

Fig. 1

202

Combine chelating organic acid buffer system and abrasive with water

204

Further combine an oxidizer

206

Further combine a corrosion inhibitor

Fig. 2

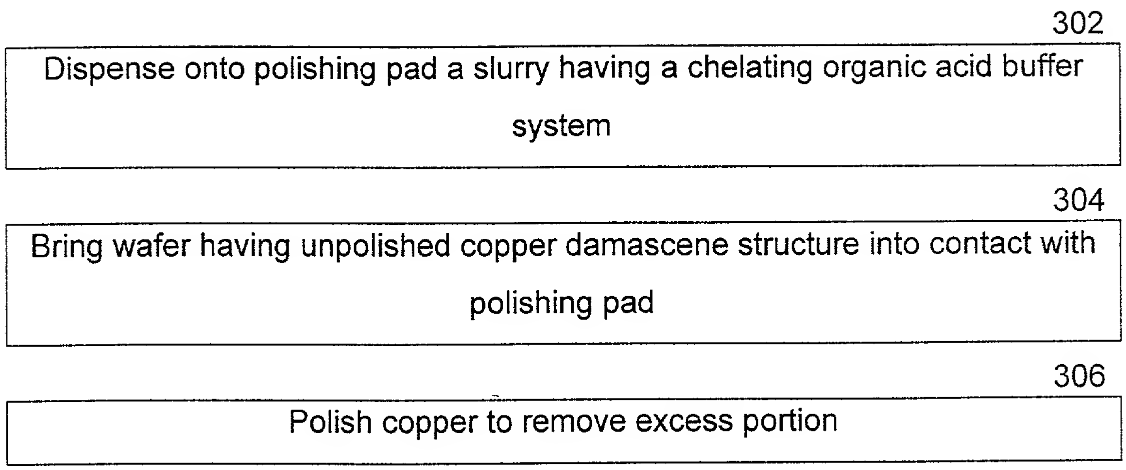
302
Dispense onto polishing pad a slurry having a chelating organic acid buffer system
304
Bring wafer having unpolished copper damascene structure into contact with polishing pad
306
Polish copper to remove excess portion

Fig. 3

402

Form a copper diffusion barrier layer over patterned ILD layer

404

Form a copper seed layer over copper diffusion barrier layer

406

Form a copper layer over the barrier and seed layers

408

Remove excess portion of copper layer by CMP with first slurry that includes a chelating organic acid buffer system and an oxidizer

410

Remove excess portion of copper diffusion barrier layer by CMP with second slurry that includes a chelating organic acid buffer system and excludes an oxidizer

Fig. 4

DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION
(FOR INTEL CORPORATION PATENT APPLICATIONS)

As a below named inventor, I hereby declare that:

My residence, mailing address and citizenship are as stated below, next to my name.

I believe I am the original, first, and sole inventor (if only one name is listed below) or an original, first, and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

SLURRY AND METHOD FOR CHEMICAL MECHANICAL POLISHING OF COPPER

the specification of which

[X] is attached hereto.
[ ] was filed on ____________ as
United States Application Number ____________
or PCT International Application Number ____________
and was amended on ____________.
(if applicable)

ereby state that I have reviewed and understand the contents of the above-identified specification, including the claim(s), as iended by any amendment referred to above. I do not know and do not believe that the claimed invention was ever known or ed in the United States of America before my invention thereof, or patented or described in any printed publication in any untry before my invention thereof or more than one year prior to this application, that the same was not in public use or on le in the United States of America more than one year prior to this application, and that the invention has not been patented or ade the subject of an inventor's certificate issued before the date of this application in any country foreign to the United States America on an application filed by me or my legal representatives or assigns more than twelve months (for a utility patent plication) or six months (for a design patent application) prior to this application.

acknowledge the duty to disclose all information known to me to be material to patentability as defined in Title 37, Code of ederal Regulations, Section 1.56.

hereby claim foreign priority benefits under Title 35, United States Code, Section 119(a)-(d), of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s):

APPLICATION NUMBER	COUNTRY (OR INDICATE IF PCT)	DATE OF FILING (day, **month**, year)	PRIORITY CLAIMED UNDER 37 USC 119
			[ ] No [ ] Yes
			[ ] No [ ] Yes
			[ ] No [ ] Yes

I hereby claim the benefit under Title 35, United States Code, Section 119(e) of any United States provisional application(s) listed below:

APPLICATION NUMBER	FILING DATE

I hereby claim the benefit under Title 35, United States Code, Section 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, Section 112, I acknowledge the duty to disclose all information known to me to be material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

APPLICATION NUMBER	FILING DATE	STATUS (ISSUED, PENDING, ABANDONED)

I hereby appoint the persons listed on Appendix A hereto (which is incorporated by reference and a part of this document) as my respective patent attorneys and patent agents, with full power of substitution and revocation, to prosecute this application and to transact all business in the Patent and Trademark Office connected herewith.

Send correspondence to:
Raymond J. Werner, Reg. No. 34,752, BLAKELY, SOKOLOFF, TAYLOR & ZAFMAN, LLP
(Name of Attorney or Agent)
12400 Wilshire Boulevard, 7th Floor, Los Angeles, California 90025 and direct telephone calls to:
ymond J. Werner, (503) 684-6200.
ame of Attorney or Agent)

ereby declare that all statements made herein of my own knowledge are true and that all statements made on information and lief are believed to be true; and further that these statements were made with the knowledge that willful false statements and e like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and at such willful false statements may jeopardize the validity of the application or any patent issued thereon.

'ull Name of Sole/First Inventor (given name, family name) **Anne E. Miller**

nventor's Signature ______________________ Date ______________

Residence ______________________ Citizenship ______________
(City, State) (Country)

P. O. Address ______________________________________

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Full Name of Second/Joint Inventor (given name, family name) **A. Daniel Feller**

Inventor's Signature ______________________ Date ____________

Residence ______________________ Citizenship ____________
(City, State) (Country)

P. O. Address ______________________

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Full Name of Third/Joint Inventor (given name, family name) **Kenneth C. Cadien**

Inventor's Signature ______________________ Date ____________

Residence ______________________ Citizenship ____________
(City, State) (Country)

O. Address ______________________

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ull Name of Fourth/Joint Inventor (given name, family name) ____________

ventor's Signature ______________________ Date ____________

esidence ______________________ Citizenship ____________
(City, State) (Country)

. O. Address ______________________

____

Full Name of Fifth/Joint Inventor (given name, family name) ____________

Inventor's Signature ______________________ Date ____________

Residence ______________________ Citizenship ____________
(City, State) (Country)

P. O. Address ______________________

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APPENDIX A

William E. Alford, Reg. No. 37,764; Farzad E. Amini, Reg. No. 42,261; William Thomas Babbitt, Reg. No. 39,591; Carol F. Barry, Reg. No. 41,600; Jordan Michael Becker, Reg. No. 39,602; Lisa N. Benado, Reg. No. 39,995; Bradley J. Bereznak, Reg. No. 33,474; Michael A. Bernadicou, Reg. No. 35,934; Roger W. Blakely, Jr., Reg. No. 25,831; R. Alan Burnett, Reg. No. 46,149; Gregory D. Caldwell, Reg. No. 39,926; Andrew C. Chen, Reg. No. 43,544; Thomas M. Coester, Reg. No. 39,637; Donna Jo Coningsby, Reg. No. 41,684; Florin Corie, Reg. No. 46,244; Dennis M. deGuzman, Reg. No. 41,702; Stephen M. De Klerk, Reg. No. P46,503; Michael Anthony DeSanctis, Reg. No. 39,957; Daniel M. De Vos, Reg. No. 37,813; Sanjeet Dutta, Reg. No. P46,145; Matthew C. Fagan, Reg. No. 37,542; Tarek N. Fahmi, Reg. No. 41,402; George Fountain, Reg. No. 37,374; James Y. Go, Reg. No. 40,621; James A. Henry, Reg. No. 41,064; Willmore F. Holbrow III, Reg. No. P41,845; Sheryl Sue Holloway, Reg. No. 37,850; George W Hoover II, Reg. No. 32,992; Eric S. Hyman, Reg. No. 30,139; William W. Kidd, Reg. No. 31,772; Sang Hui Kim, Reg. No. 40,450; Walter T. Kim, Reg. No. 42,731; Eric T. King, Reg. No. 44,188; Erica W. Kuo, Reg. No. 42,775; George B. Leavell, Reg. No. 45,436; Gordon R. Lindeen III, Reg. No. 33,192; Jan Carol Little, Reg. No. 41,181; Kurt P. Leyendecker, Reg. No. 42,799; Joseph Lutz, Reg. No. 43,765; Michael J. Mallie, Reg. No. 36,591; Andre L. Marais, under 37 C.F.R. § 10.9(b); Paul A. Mendonsa, Reg. No. 42,879; Clive D. Menezes, Reg. No. 45,493; Chun M. Ng, Reg. No. 36,878; Thien T. Nguyen, Reg. No. 43,835; Thinh V. Nguyen, Reg. No. 42,034; Dennis A. Nicholls, Reg. No. 42,036; Daniel E. Ovanezian, Reg. No. 41,236; Kenneth B. Paley, Reg. No. 38,989; Gregg A. Peacock, Reg. No. 45,001; Marina Portnova, Reg. No. P45,750; William F. Ryann, Reg. 44,313; James H. Salter, Reg. No. 35,668; William W. Schaal, Reg. No. 39,018; James C. Scheller, Reg. No. 31,195; Jeffrey Sam Smith, Reg. No. 39,377; Maria McCormack Sobrino, Reg. No. 31,639; Stanley W. Sokoloff, Reg. No. 25,128; Judith A. Szepesi, Reg. No. 39,393; Vincent P. Tassinari, Reg. No. 42,179; Edwin H. Taylor, Reg. No. 25,129; John F. Travis, Reg. No. 43,203; Joseph A. Twarowski, Reg. No. 42,191; Thomas A. Van Zandt, Reg. No. 43,219; Lester J. Vincent, Reg. No. 31,460; Glenn E. Von Tersch, Reg. No. 1,364; John Patrick Ward, Reg. No. 40,216; Mark L. Watson, Reg. No. P46,322; Thomas C. Webster, Reg. No. P46,154; and Norman Zafman, Reg. No. 26,250; my patent attorneys, and Justin M. Dillon, Reg. No. 42,486 and Raul Martinez, Reg. No. 46,904, my patent agents; of BLAKELY, SOKOLOFF, TAYLOR & ZAFMAN LLP, with offices located at 12400 Wilshire Boulevard, 7th Floor, Los Angeles, California 90025, telephone (310) 207-3800, and Alan K. Aldous, Reg. No. 1,905; Robert D. Anderson, Reg. No. 33,826; Joseph R. Bond, Reg. No. 36,458; Richard C. Calderwood, Reg. No. 35,468; Jeffrey S. Draeger, Reg. No. 41,000; Cynthia Thomas Faatz, Reg No. 39,973; Sean Fitzgerald, Reg. No. 32,027; John N. Greaves, Reg. No. 40,362; Seth Z. Kalson, Reg. No. 40,670; David J. Kaplan, Reg. No. 41,105; Charles A. Mirho, Reg. No. 41,199; Leo V. Novakoski, Reg. No. 37,198; Naomi Obinata, Reg. No. 39,320; Thomas C. Reynolds, Reg. No. 32,488; Kenneth M. Seddon, Reg. No. 43,105; Mark Seeley, Reg. No. 32,299; Steven P. Skabrat, Reg. No. 36,279; Howard A. Skaist, Reg. No. 36,008; Steven C. Stewart, Reg. No. 33,555; Raymond J. Werner, Reg. No. 34,752; Robert G. Winkle, Reg. No. 37,474; Steven D. Yates, Reg. No. 42,242, and Charles K. Young, Reg. No. 39,435; my patent attorneys, and Thomas Raleigh Lane, Reg. No. 42,781; Calvin E. Wells; Reg. No. P43,256, Peter Lam, Reg. No. 44,855; and Gene I. Su, Reg. No. 45,140; my patent agents, of INTEL CORPORATION; and James R. Thein, Reg. No. 31,710, my patent attorney; with full power of substitution and revocation, to prosecute this application and to transact all business in the Patent and Trademark Office connected herewith.

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SLURRY AND METHOD FOR CHEMICAL MECHANICAL POLISHING OF COPPER

the specification of which

☐ is attached hereto.
☒ was filed on **November 16, 2001** as
United States Application Number **09/715,282**
or PCT International Application Number ________
and was amended on ________.
(if applicable)

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claim(s), as amended by any amendment referred to above. I do not know and do not believe that the claimed invention was ever known or used in the United States of America before my invention thereof, or patented or described in any printed publication in any country before my invention thereof or more than one year prior to this application, that the same was not in public use or on sale in the United States of America more than one year prior to this application, and that the invention has not been patented or made the subject of an inventor's certificate issued before the date of this application in any country foreign to the United States of America on an application filed by me or my legal representatives or assigns more than twelve months (for a utility patent application) or six months (for a design patent application) prior to this application.

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			☐ No ☐ Yes
			☐ No ☐ Yes
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(Name of Attorney or Agent)
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(Name of Attorney or Agent)

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Full Name of Fourth/Joint Inventor (given name, family name)

Inventor's Signature Date

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Full Name of Fifth/Joint Inventor (given name, family name)

Inventor's Signature Date

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